

# Influences of Silicabeads on the Venting of Weakly and Strongly Foaming Systems

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## Abstract

Venting of systems containing solids has so far been treated as venting of two-phase systems and the presence of particles has been neglected. For nonfoaming systems, this has shown to be an appropriate assumption. To verify if this assumption is also valid for foaming systems, experiments were carried out regarding venting foaming systems without particles and in presence of silicabeads of varying sizes. Results show that particles influence the venting behaviour. These results differ largely in respect to the degree of the foaminess of the system. Nevertheless recommendations for the design of pressure relief devices for foaming two-phase systems are with some limitations also applicable for three-phase systems.

## Keywords

Venting; Foam; Multi-phase

## Motivation and Objectives

Phenomena during vent processes have been investigated for several decades. [DIERS, 1986] Starting with single-phase flow design models have now evolved to a precise design of venting devices for two-phase flow [Schmidt, 2007]. Nevertheless, highly viscous systems and foaming systems still lead to limitations that can only be met by over conservative assumptions. [Duh, Hu, Chang, Kao, 2009]

Addition to liquid and vapour components solids inside the systems vented becomes in terms of heterogeneous catalysis more important. For nonfoaming systems, research showed that they do not have a significant impact on the venting behaviour. [Waldram, McIntosh, Etchells, 2006] Multiple variations of particle parameters such as size, density and surface structure did not lead to significant differences in the venting behaviour. Design of nonfoaming systems containing solids is thus treated as if the particles were not present. [Poli, Imhof, Holst, Steinbach, 2008]

The influence of solids on the venting of foaming systems has not been deeply investigated so far. It has

been shown that two different scenarios can occur. Either the particles can be vented almost completely at the beginning of the venting process or they can be hardly vented at all. Apart from those phenomenological observations, no direct influence has been described so far thus leading to problems in the design of venting devices for those scenarios.

To determine how solids affect the venting behaviour venting experiments were carried out weakly for strongly foaming systems with and without the presence of particles. This research will lead to more precise recommendations for the design of venting devices for foaming three-phase systems.

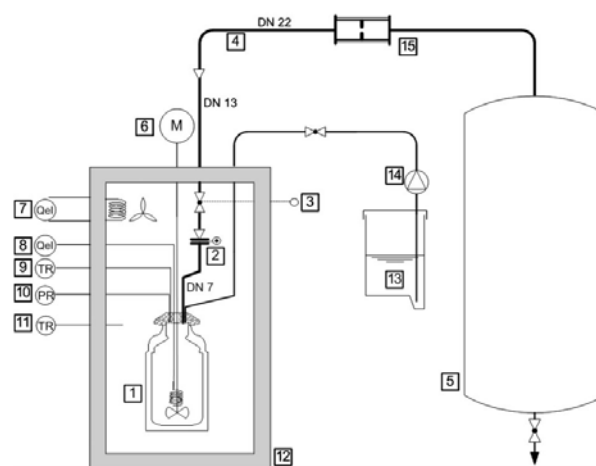


FIG. 1 SCHEMATIC SETUP OF THE ADCII TAKEN OUT OF POLI, IMHOF, HOLST, STEINBACH [Poli, Imhof, Holst, Steinbach, 2008]:

(1) Reactor (1.2 L); (2) Orifice (2 mm); (3) Pneumatic driven ball valve; (4) Venting pipe; (5) 120 L-catch tank; (6) Stirrer; (7) Heater; (8) Heatercoil; (9) Temperature transducer; (10) Pressure transducer; (11) Temperature transducer; (12) Oven; (13) Feed supply; (14) Peristaltic pump; (15) Glass tube with orifice (optional)

## Experimental

### Experimental Setup

Experiments were carried out in a modified ADCII, Adiabatic Dewar Calorimeter II, by Chilworth Technology, Inc. The system consisted of a 1.2l reactor with a height to diameter ratio of roughly 4:1. The top

and bottom of the reactor were constructed with steel while the walls were made of glass to get an inside view on the phenomena occurring during venting. A detailed description can be found in FIG. 1.

The systems were heated internally via a heatercoil. An oven surrounded the reactor following the temperature of the reactor and thus reducing energy losses of the reactor to the surrounding to a minimum.

Venting was initiated by opening a ball valve with a pneumatic lift. The ventline was then limited to a minimal diameter of 2 mm at an orifice. During heatup, venting pressure and temperature of the reactor as well as the temperature of the oven were recorded.

For the analysis of foam, structure and particle behaviour during venting images of the reactor were recorded at 10 Hz with the high resolution CCD-camera CV-M40 by JAI Corporation.

### Investigated Systems

Unstirred vapour pressure systems of water were investigated. To induce strong foaming sodium dodecyl sulfat, SDS, by Merck Schuchardt OHG was added to deionized water in concentrations from 0.005g/l to 0.5g/l. All experiments with SDS were prepared with a solution of 1g/l SDS that was stirred at room temperature for at least 48 h before use since SDS hydrolizes forming an alcohol that increases the foaminess. To prove that after 48 h no influence of the ageing is seen, venting experiments for solutions left ageing for up to one month were conducted. Results showed no significant differences in their venting behaviour. For weakly foaming systems isobutanol was added to deionized water in concentrations of 1w% to 50w%.

As solids, Silicabeads Typ S by Sigmund Lindner GmbH were choosen. They consist mostly of  $\text{SiO}_2$ . Their shape is that of a perfect ball with a plain surface. Size fractions of 0-50  $\mu\text{m}$ , 70-140  $\mu\text{m}$  and 90-150  $\mu\text{m}$  were used to study the effect of differing particle weights. For the three-phase experiments conducted the mass fraction of the particles to the overall system was kept at 20w%.

All systems investigated had a fill level of 85%. The venting pressure was kept constant for all experiments at 6 bar.

## Results

### Strongly Foaming Systems

Venting of strongly foaming systems showed a strong

influence of the particles on the venting behaviour. At low SDS-concentrations, minor effects of the particles on the pressure decrease over time was observed.

For experiments with 0.005g/l SDS up to 2.5 bar, no significant differences were seen for all two-phase and three-phase systems. At pressures below 2.5 bar, the pressure decrease of systems containing particles is lower than that of the two-phase sytems.. The medium sized and large particles behaved very similar and showed a maximum deviation to the two-phase system of roughly 0.1 bar. The difference for the systems with the small particles was more pronounced. Differences of up to 0.3 bar in comparison to the two-phase systems were observed. Pressure over time for the experiments at 0.005g/l SDS is shown in FIG. 2.

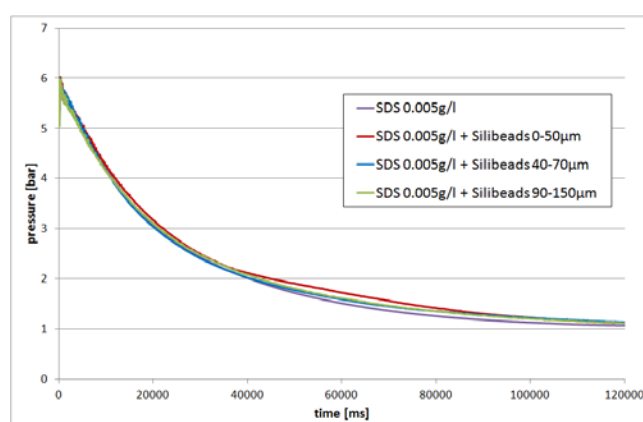


FIG. 2 PRESSURE OVER TIME FOR EXPERIMENTS CONTAINING 0.005g/l SDS

The differences of the behaviour of the small particles in comparison to other particles can be explained by the degree of suspension of the particles in the reactor. While the larger particles stay at the bottom of the reactor throughout the whole vent process, the small particles are lifted up by the bubbles forming at the bottom of the reactor and within the particles.

At the beginning of the experiment, the small particles also lay as a stable layer at the bottom. But over time more and more particles are suspended until at roughly 2.5 bar all particles are suspended within the water. At this point, the foam structure dramatically changes. The bubble size inside the foam shrinks appears to be more stable.

This can be explained by the size of bubbles reaching the surface. Due to the layer of particles at the bottom of the reactor, small bubbles formed cannot rise upon formation. They have to coalescend with other bubbles and grow in size until their buoyant force is strong enough to break through the particles holding them down. Once all particles are suspended, smaller

bubbles can rise and thus changing the structure of the foam.

For the other particles, massive eruptions of large bubbles are observed especially at lower pressures. Those eruptions rupture the foam structure above the liquid. Since the foam structure of SDS is relatively stable, not all of the foam is destroyed and two-phase discharge continues.

At the end of the experiment, the foam collapses and only single-phase discharge is observed from then on. For all experiments with SDS, this occurred at roughly 1.4 bar

At higher concentrations of SDS, the behaviour of two-phase and three-phase systems differs more significantly. For concentrations larger than 0.05g/l pressure decrease over time is faster when the concentration of SDS is increased. This is not the same for systems containing particles. Their pressure decrease over time stagnates in this range of concentrations. As an example, pressure over time for systems containing 0.07g/l is presented in FIG. 3.

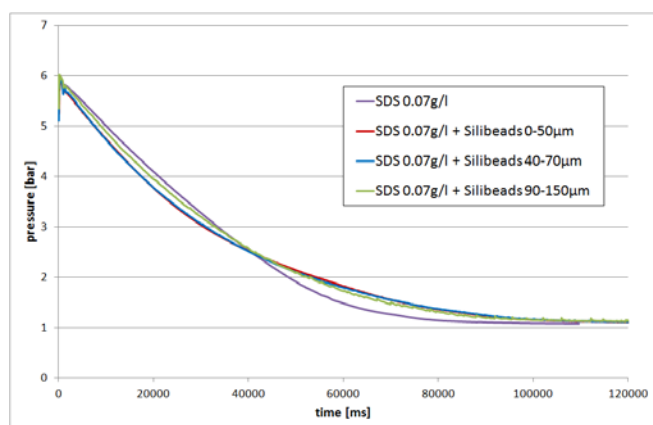


FIG. 3 PRESSURE OVER TIME FOR EXPERIMENTS CONTAINING 0.07g/l SDS

Explanations for this behaviour can again be found in the foam structure during the experiments. Due to an unrestricted bubble rise in two-phase systems, very dry foam structures are established with a low liquid mass fraction and thin boundary layers. Thus more gas is vented than that in the three-phase systems and pressure decrease is enhanced.

Addition to the pressure over time during venting the mass discharge was analysed. Liquid and solid were analyzed independently. All information is given as relative discharge, meaning discharge of the solids or liquid over their initial mass at the beginning of the experiment.

For the relative liquid discharge of systems containing

SDS, as shown in FIG. 4, a clear effect of the particles especially at low concentrations is found. At 0.005g/l SDS, the discharge is more than tripled by the addition of the smallest particles. For scenarios taking impurities or leftovers from cleaning into account, this effect may lead to dramatic increase of size and thus the cost of downstream components like catch vessels or gas separators has to be taken into account during design.

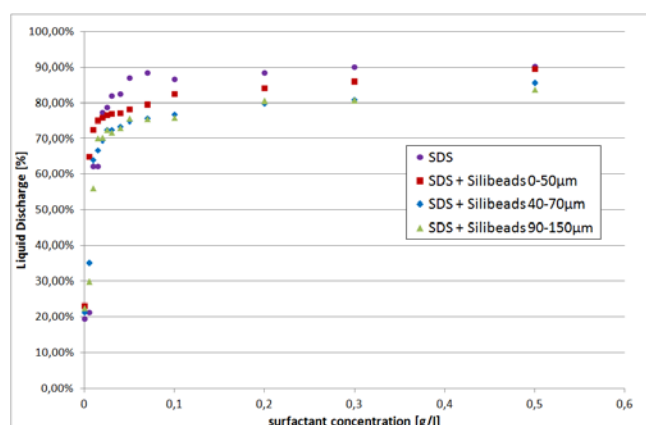


FIG. 4 RELATIVE OVERALL DISCHARGE OF LIQUID FOR SYSTEMS CONTAINING SDS

At SDS concentrations larger than 0.02g/l, the mass discharge of the two-phase systems exceeds that of the three-phase systems. Differences are not larger than 15% and in a range where almost complete discharge of the liquid is observed. Thus the implications are not that significant as for the effect at low SDS concentrations.

Compared with the three-phase systems, increased mass discharge with decreasing particle size is observed. The difference of the small particles is more pronounced than that of the medium sized and large particles. This is explained by the more stable foam structure at the end on the vent process as described before.

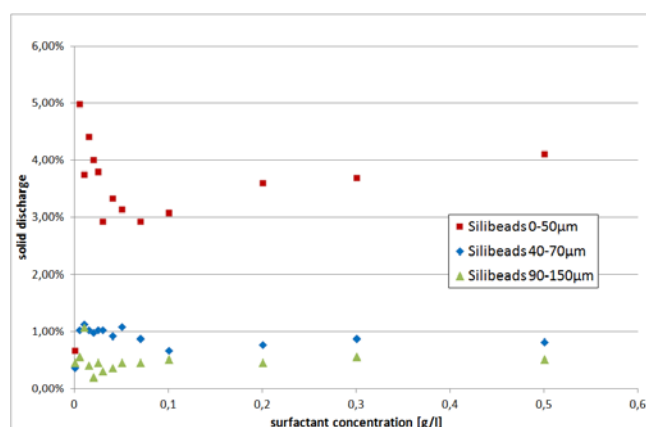


FIG. 5 RELATIVE OVERALL DISCHARGE OF SOLIDS FOR SYSTEMS CONTAINING SDS

Solid discharge is hardly observed. A maximum of 5% of the solids is discharged during the vent process. This equals 1% of the overall mass. Thus solid discharge can be neglected in the design of venting devices for the particles investigated. As shown in FIG.5, the particle discharge increases with decreasing particle size. Only around 1% of the medium sized particles and large particles was vented.

### Weakly Foaming Systems

The effect of particles on the venting of weakly foaming systems was quite different from that on the strongly foaming systems. At low concentrations particles slow the pressure decrease over time. As shown in FIG. 6 for systems containing 3w% isobutanol, the pressure of two-phase systems exceeds that of three-phase systems by up to 0.5 bar till a pressure of 2 bar.

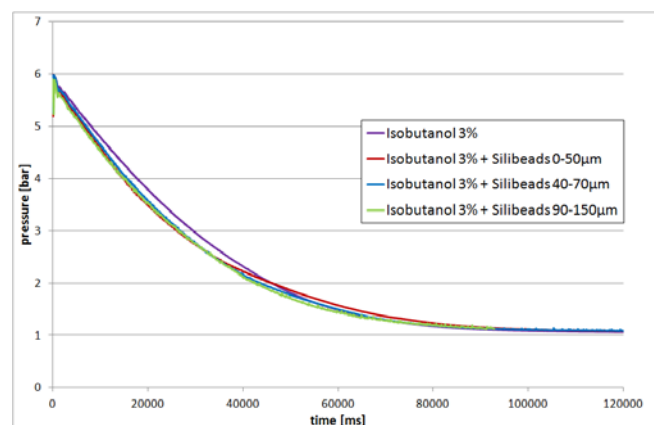


FIG. 6 PRESSURE OVER TIME FOR EXPERIMENTS CONTAINING 3w% ISOBUTANOL

Differences can again be seen in the venting of the three different particles sizes. Again the smallest particles show a different behaviour from the other particles. As in the case of strongly foaming systems they form a homogenous suspension at roughly 2.5 bar and foam structure changes.

The larger particles form a layer at the bottom of the reactor letting only large bubbles pass through. The effect of those large bubbles on the foam structure of the weakly foaming systems is similar to the effect described for the strongly foaming systems. The large bubbles rupture the foam layer on top of the liquid. But in the case of weakly foaming systems, almost all of the foam is destroyed and discharge turns single-phase for a short period of time till smaller bubbles rebuild the foam. The destructions are more pronounced for the largest particles. The process of the foam rupture by large bubbles and the resulting single-phase discharge as observed for the medium

sized and large particles can be seen in FIG. 7.

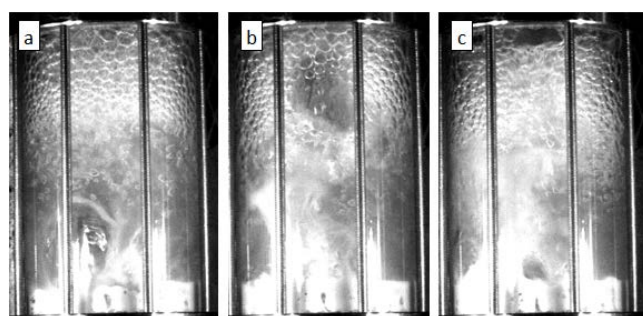


FIG. 7 RISE OF A LARGE BUBBLE THROUGH THE PARTICLE LAYER (a), FOAM RUPTURE (b) AND RESULTING SINGLE-PHASE DISCHARGE (c) AS OBSERVED DURING THE VENTING OF A SYSTEM CONTAINING 3w% ISOBUTANOL AND SILICABEADS 90-150µm

As the concentration of isobutanol is increased, the behaviour of the systems remains similar with the difference between the two-phase and three-phase systems increase as can be seen in FIG. 8 for systems containing 10w% isobutanol.

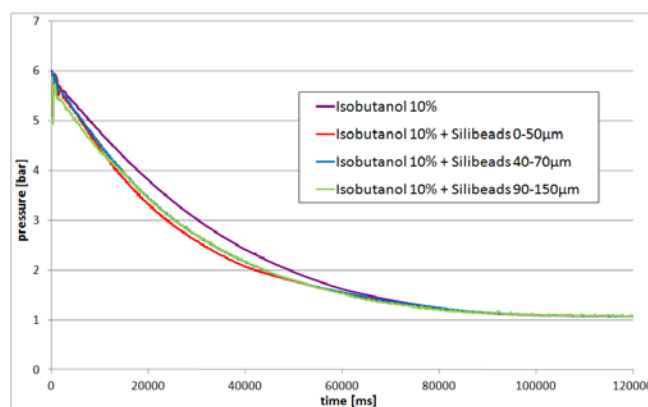


FIG. 8 PRESSURE OVER TIME FOR EXPERIMENTS CONTAINING 10w% ISOBUTANOL

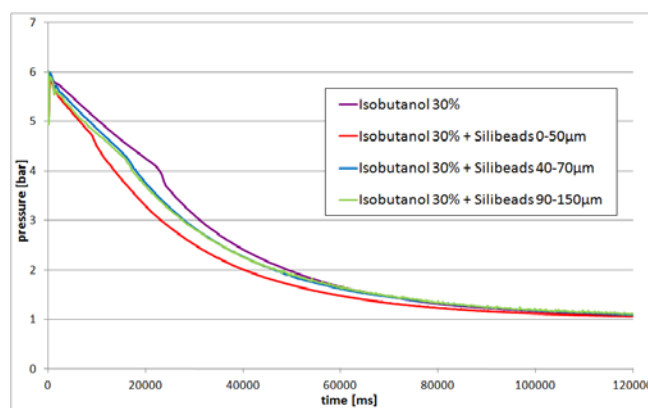


FIG. 9 PRESSURE OVER TIME FOR EXPERIMENTS CONTAINING 30w% ISOBUTANOL

At concentrations of isobutanol that exceed the solubility limit of isobutanol in water for the temperature range of 100°C-160°C observed during the vent process, an interesting effect occurred. For experiments containing 30w% isobutanol, an abrupt

destruction of the foam was observed at 4.0 bar to 4.5 bar. From the moment of destruction, no formation of foam was observed for all experiments even though a large amount of bubbles was still formed during the rest of the vent process. The pressure over time for those experiments can be found in FIG. 9.

This effect is explained by an excess layer of isobutanol that forms on the surface of the liquid. Once it reaches a certain thickness, it destabilizes the surface of the bubbles formed since no foam can be formed within a pure substance [Wasan, Nikolov, Shah, 2004].

Surprisingly, this effect was not found for systems containing 50w% isobutanol. For the two-phase system as well as the three-phase systems containing 50w% isobutanol, foam was observed throughout the whole experiment. Seemingly, only a specific range of concentrations meets the conditions in which the foam structure is unstable enough to be destroyed within the isobutanol layer. For 50w% isobutanol, the foam structure may already be too stable to be ruptured during the short passage through the excess layer.

The mass discharge of the systems containing isobutanol followed the trends observed for the pressure decrease over time as shown in FIG. 10.

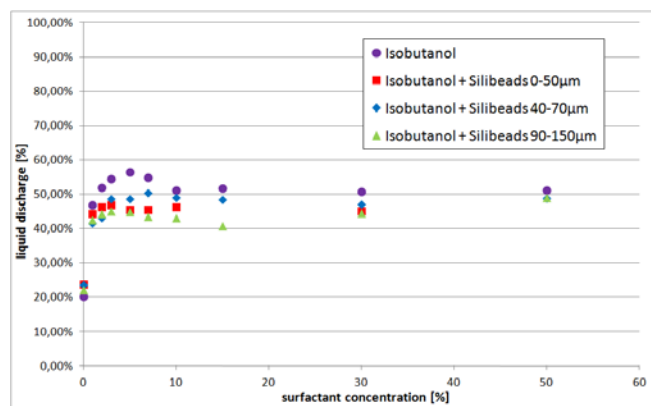


FIG. 10 RELATIVE OVERALL DISCHARGE OF LIQUID FOR SYSTEMS CONTAINING ISOBUTANOL

For all systems investigated, the mass discharge of liquid ranges between 40% and 60%. The discharge of two-phase systems exceeds that of all three-phase systems for all concentrations investigated. Differences range up to 10%. Surprisingly, the mass discharge of the experiments with 30w% isobutanol is not less than that of the other experiments. Most of the mass discharge seems to occur at the beginning of the vent process. Thus the break down of the foam structure does not affect the discharge significantly.

The mass discharge of solids for weakly foaming

systems was similar to that of strongly foaming systems as can be seen in FIG. 11. A maximum of 3.2% of the particles were vented. Again the smallest particles were discharged significantly more than the medium sized and large particles.

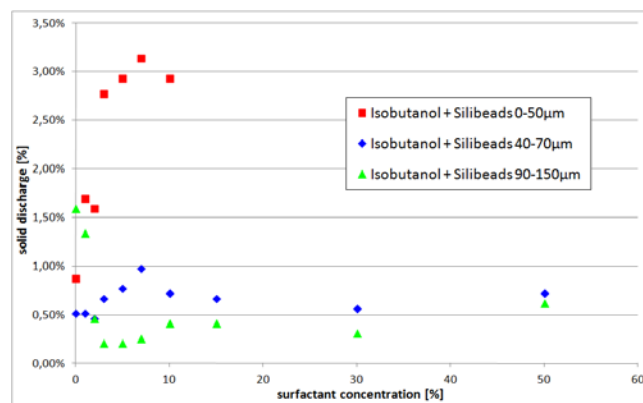


FIG. 11 RELATIVE OVERALL DISCHARGE OF SOLIDS FOR SYSTEMS CONTAINING ISOBUTANOL

## Conclusions and Recommendations

Experiments showed that the addition of particles to foaming systems affects the vent process in multiple ways. Particle addition leads to a prolonged two-phase discharge if foams are stable enough to endure the turbulences created by the particles inside the reactor.

Thus strongly foaming systems that have very stable foam structures have an increased mass discharge by the addition of particles and pressure decrease over time is slowed. In contrast, for weakly foaming systems where instable foam is observed, decrease of the overall mass discharge and faster pressure decrease over time was found.

The main reason for the increased two-phase discharge for stable foams can be found in a general increase of the foaminess by addition of particles. Experiments carried out in another setup showed that the particles function as nucleation point and induce a more evenly distributed bubble formation over the crosssection of the reactor. [Leimeister, Steinbach (a), 2013] This effect is more pronounced at low concentrations since at higher concentrations two-phase mass discharge is observed throughout the complete vent process.

In the systems investigated in this paper, no significant discharge of particles was found. Previous experiments showed scenarios with almost complete particle discharge. [Leimeister, Steinbach (b), 2013]. The major parameter to explain these different behaviours seems to be the hydrophilic properties of



the particles. In aqueous systems, hydrophobic particles are vented almost completely while hydrophilic particles are hardly vented at all. Recommendations given in this paper are limited to scenarios of aqueous systems with hydrophilic particles as third phase.

For the systems investigated in this paper, the use of the recommendations of Schecker [Schecker, Friedel, 2004] is suggested. Schecker experimentally determined the radial distribution parameter during vent processes for weakly and strongly foaming systems. It was concluded that for the radial distribution parameter of 1.01 for strongly and 1.2 for weakly foaming systems, the use of the model by Henry-Fauske with a submodel of DIERS bubbly flow in the reactor is suggested for the design of venting devices. [Schecker, 2003]

For weakly foaming systems this will also lead to conservative results for three-phase systems. This accounts for low concentrations as well as for oversaturated systems.

For strongly foaming systems, the recommendation is also valid under the restriction that low concentrations are also treated as if they would lead to constant two-phase discharge. This may be over conservative, but since the mass discharge differs so significantly between two-phase and three-phase systems, no other option seems to be valid. The effect of the particles on the pressure decrease over time can be neglected for the design since the major differences occur at pressures where the danger for the reactor is not that serious.

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**Prof. Jörg Steinbach** has earned a diploma in Chemistry from the Technische Universität Berlin, Germany in 1981. He afterwards did his PhD in Chemistry at the Technische Universität Berlin, Germany in 1985. While being employed at ScheringAG, Germany from 1985-1995 he achieved his habilitation in safety engineering in 1994. Since 1996 he has

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Prof. Steinbach is chairman of the board of ASIIN e.V., member of the Presidents Advisory Council of the

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